

Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/US2005/003264

International filing date: 03 February 2005 (03.02.2005)

Document type: Certified copy of priority document

Document details: Country/Office: US
Number: 60/566,699
Filing date: 30 April 2004 (30.04.2004)

Date of receipt at the International Bureau: 01 December 2006 (01.12.2006)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)



World Intellectual Property Organization (WIPO) - Geneva, Switzerland
Organisation Mondiale de la Propriété Intellectuelle (OMPI) - Genève, Suisse



THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

UNITED STATES DEPARTMENT OF COMMERCE

United States Patent and Trademark Office

November 20, 2006

THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM THE RECORDS OF THE UNITED STATES PATENT AND TRADEMARK OFFICE OF THOSE PAPERS OF THE BELOW IDENTIFIED PATENT APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A FILING DATE.

APPLICATION NUMBER: 60/566,699

FILING DATE: *April 30, 2004*

RELATED PCT APPLICATION NUMBER: PCT/US05/03264

THE COUNTRY CODE AND NUMBER OF YOUR PRIORITY APPLICATION, TO BE USED FOR FILING ABROAD UNDER THE PARIS CONVENTION, IS *US60/566,699*



Certified by

Under Secretary of Commerce
for Intellectual Property
and Director of the United States
Patent and Trademark Office

22651 U.S. PTO
043004

PTO/SB/16 (01-04)
Approved for use through 07/31/2006. OMB 0651-0032
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

Express Mail Label No.

EV 398521077 US

INVENTOR(S)					
Given Name (first and middle [if any])		Family Name or Surname		Residence (City and either State or Foreign Country)	
JOHN		LOMBARDI		831 N. CAMINO MIRAMONTE TUCSON, AZ 85716, USA	
Additional inventors are being named on the _____ separately numbered sheets attached hereto					
TITLE OF THE INVENTION (500 characters max)					
LAPPING COMPOSITION AND METHOD USING SAME					
Direct all correspondence to: CORRESPONDENCE ADDRESS					
<input type="checkbox"/> Customer Number: _____					
OR					
<input checked="" type="checkbox"/> Firm or Individual Name DALE F. REGELMAN					
Address LAW OFFICE OF DALE F. REGELMAN, P.C.					
Address 4231 S. FREMONT AVENUE					
City TUCSON		State AZ		Zip	85714
Country UNITED STATES OF AMERICA		Telephone 520.741.7636		Fax	520.746.9114
ENCLOSED APPLICATION PARTS (check all that apply)					
<input checked="" type="checkbox"/> Specification Number of Pages 29					
<input type="checkbox"/> Drawing(s) Number of Sheets _____					
<input type="checkbox"/> Application Data Sheet. See 37 CFR 1.76					
<input type="checkbox"/> CD(s), Number _____					
<input type="checkbox"/> Other (specify) _____					
METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT					
<input checked="" type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27.					
<input type="checkbox"/> A check or money order is enclosed to cover the filing fees.					
<input checked="" type="checkbox"/> The Director is hereby authorized to charge filing fees or credit any overpayment to Deposit Account Number: 502262					
<input checked="" type="checkbox"/> Payment by credit card. Form PTO-2038 is attached.					
FILING FEE Amount (\$) 80.00					
The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.					
<input checked="" type="checkbox"/> No.					
<input type="checkbox"/> Yes, the name of the U.S. Government agency and the Government contract number are: _____					

Respectfully submitted

[Page 1 of 2]

Date April 30, 2004

SIGNATURE

TYPED or PRINTED NAME Dale F. Regelman

TELEPHONE (520) 741-7636

REGISTRATION NO. 45,625

(if appropriate)

Docket Number: LOMBARDI 04.01-P

USE ONLY FOR FILING A PROVISIONAL APPLICATION FOR PATENT

This collection of information is required by 37 CFR 1.51. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 8 hours to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Mail Stop Provisional Application, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

CERTIFICATE OF EXPRESS MAILING

"Express Mail" Mailing Label No. EV 398521077 US Date of Deposit April 30, 2004

I hereby certify that this paper and the papers listed thereon are being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above, and is addressed to **Mail Stop Provisional Application, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

Signature of person mailing: _____

Name of person mailing: Reena Mendez

LAPPING COMPOSITION AND METHOD USING SAME

Lapping, grinding, cutting and polishing, collectively referred to herein as “lapping,” are key manufacturing technologies for shaping various ceramic and metallic materials. A number of factors influence the quality of surfaces shaped by these methods including the size and composition of the particular abrasive grit employed as well as the amount of pressure applied to the surface. Fluids are often also cascaded upon the part during these operations. Among other functions, such fluids remove heat during machining. In addition, such fluids lubricate the abrasive / surface contact area. Such fluids also remove swarf from the part.

Rapid swarf removal is desirable in order to efficiently shape the part as well as reproducibly produce smooth, high quality surfaces. Hence it is desirable that the machining fluid exhibits a significant affinity for the surface of the material being shaped. In certain embodiments, such an affinity includes chemisorption upon the ceramic or metallic material; such that its swarf is wetted, dispersed within, and ultimately washed away by the cascading machining fluid.

The invention will be described herein as embodied in a lapping composition useful in the manufacture of read / write heads for computer hard disks. The following description of Applicant’s composition, and uses thereof, is not meant, however, to limit Applicant’s invention to the manufacture of such hard disks, or to the manufacture of data storage devices in general, as the invention herein can be applied to surface preparation of metals, ceramics, and the like. In addition, Applicant’s compositions are useful in a myriad of applications, including without limitation polishing and/or chemical

mechanical polishing of various substrates, removal of metal particles from filters disposed in waste water treatment plants, manufacture of semiconductor devices, and the like.

As one example, the manufacture of read / write heads for computer hard disks, and the manufacture of the hard disks, includes one or more lapping processes. As those skilled in the art will appreciate, such computer hard disks comprise rotatable disks comprising one or more magnetic materials. Information is written to, and read from, such magnetic disks using a read/write head.

As those skilled in the art will appreciate, computer hard disks are rotatably disposed in a disk drive unit such that the magnetic disk rotates rapidly as information is written thereto and/or read therefrom. In order to prevent the read/write head from damaging the disk surface when that disk is rapidly rotating, it is critical that the read/write head be planarized so that it exhibits minimal surface roughness.

There is an on-going need to maximize the storage density in such computer hard disks. In order to increase that storage density, the fly height between the rotating disk and the read write head is continually decreasing. As a result, increased demands are being placed upon surface quality of both the read – write head and the hard disk.

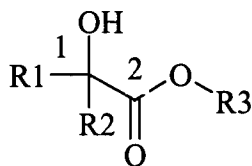
One such read/write head is often referred to as a Giant Magnetoresistive (GMR) Head. Such GMR Heads comprise a metallized ceramic material. GMR Heads typically comprise TiC – Alumina ceramic, sometimes called “AlTiC,” that has been metallized with a variety of ferrous and non-ferrous alloy layers. Prior art GMR Head lapping methods utilize fluids comprising a diamond abrasive dispersed in a mixture of water,

various alcohols, and other additives. In order to prevent corrosion of its GMR Head metal layers which would adversely effect the Head's electromagnetic properties and operational performance within the computer hard drive.

These prior art GMR Head lapping methods are inefficient, exhibiting low AlTiC
5 and metallized layer material removal rates (MRR). A low MRR is undesirable from a manufacturing standpoint because the MRR often comprises a rate-limiting step in the production of read/write heads. In addition, such prior art lapping fluids are highly alkaline, i.e. pH > 10. Moreover, these prior art lapping compositions are not biodegradable. The alkalinity in combination with the non-biodegradability of these prior
10 art compositions results in significant handling and disposal costs and concerns.

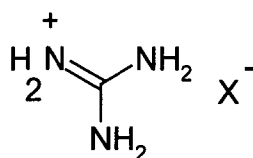
In contrast, Applicants' lapping composition is derived from natural products and are biodegradable. In addition, Applicants' lapping composition poses minimal environmental concerns. Moreover, Applicants' lapping method using Applicants' lapping fluids has a high MRR rate. Applicants' lapping composition is water-based, and
15 includes additives comprising a combination of hydroxyl and carboxylic acid / carboxylate functionalities.

In certain embodiments, Applicants' lapping composition includes salts of alpha hydroxy carboxylic acids (AHC). AHC comprise compounds having structure I, which comprises one or more hydroxyl groups attached to a first carbon atom, where that first
20 carbon atom is covalently bonded to a second carbon atom comprising a carboxyl group.

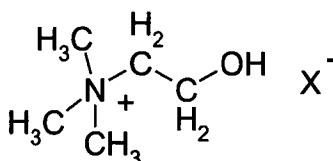


I

In certain embodiments, Applicants' lapping composition includes one or more of amine and / or alkali neutralized salts of citric, glycolic, and / or tartaric acid. In these citric acid embodiments, R1 is CH₂-CO₂-R4, and R2 is CH₂ - CO₂-R5. In these glycolic acid embodiments, R1 is methyl and R2 is hydrogen. In these tartaric acid embodiments, R1 is hydrogen and R2 is C(H)(OH)-CO₂-R6. In these embodiments, R3, R4, R5, and R6, are each independently selected from the group consisting of hydrogen, an ammonium salt, such as tetramethylammonium hydroxide, an alkali metal ion, and alkaline earth metal ion, a guanidinium cation IV, a choline cation V, and the like.



IV



V

In certain embodiments, Applicant has discovered that lapping compositions comprising Citric acid show enhanced lapping MRR performance. Table I, below, summarizes MRR using Citric acid based lapping compositions. Lapping was conducted using a Crane Lapmaster Model 15 lapping machine comprising a 15 inch diameter
5 diamond lapping film adhered to the lapping platen.

Table I.

MRR Lapping Performance of Various Citrate Salts

Formulation	Component	Solution pH	Avg. MRR (g / min.)	MRR Relative to CONTROL
A	DGA Citrate Tribasic	8.94	0.00121	303%
B	DGA Citrate Dibasic	5.06	0.00138	345%
C	MEA Citrate Tribasic	8.5	0.00135	338%
D	MEA Citrate Dibasic	5.23		
E	MEA Citrate Tribasic / Na Polyaspartate Blend	8.8	0.00122	305%
F	Na Polyaspartate	8.1	0.00095	238%
G	Na Citrate Tribasic	8.2	0.00142	355%
H	TEA Citrate Tribasic	6.8	0.00101	253%
I	TEA Citrate Tribasic / Na Polyaspartate Blend	5.05	0.00127	318%
J	TMAH Citrate Dibasic	5.19	0.00137	343%
K	MEA Citrate Tribasic Poly Sodium Aspartate – co- diglycol Aspartamide	8.8	0.00147	368%
CONTROL	Control Conventional GMR Head Lapping Fluid	10	0.00040	--

“DGA” comprises Diglycolamine. “MEA” comprises monoethanolamine. “TEA”

comprises triethanolamine. “TMAH” comprises Tetramethylammonium Hydroxide. Na

Polyaspartate was Baypure DS 100 product supplied by Bayer Corp. (Pittsburgh, PA) having approximately 1500 g/mol. molecular weight

Poly Sodium Aspartate –co- diglycol Aspartamide was prepared via aminolysis ring opening reaction of succinimide repeat units within 3000 MW Polysuccinimide (PSI) (Bayer Polymers Pittsburgh, PA) dissolved within anhydrous DMSO solvent. Solutions having < 30 wt. % 3000 MW PSI solute are desirable due to apparent PSI solubility & solution viscosity limitations. Diglycolamine was added dropwise to the PSI / DMSO solution at room temperature accompanied by rapid stirring to produce a dark red colored solution. Suitable lapping fluid aspartamide copolymers should be prepared via reaction of between 0 and 75 mol. % of PSI succinimide repeat units with diglycolamine. The remaining, unreacted SI repeat units in the aspartamide copolymer are hydrolyzed via reaction with aqueous NaOH solution in 1;1 molar stoichiometry producing the desired Poly Sodium Aspartate – co- Diglycol Aspartamide copolymer.

Formulations A through K, inclusive, and the Control, had identical Citrate Molality within Distilled H₂O, namely 0.214 Moles Citrate / Kg H₂O solvent. Formulations A through K, inclusive, were prepared via dissolution of anhydrous citric acid (A.C.S. Reagent) in distilled water followed by dropwise addition of amines accompanied by rapid stirring until a product having desired stoichiometry was obtained. Because TMAH reagent was used in its pentahydrate crystalline salt form, this material was first dissolved in distilled water prior to its addition to aqueous citric acid solution. Sodium citrate tribasic (A.C.S. Reagent) was purchased from Aldrich Chemical Company (Milwaukee, WI).

As those skilled in the art will appreciate, dissolution of anhydrous citric acid was highly endothermic, producing a solution having a temperature below about 25 oC, i.e. room temperature. Such an endothermic dissolution is advantageous because the chilled solution produced compensated for the exotherm produced from the subsequent citric acid neutralization.

As those skilled in the art will appreciate, the pKa values for the various amines used follows the order of:

TMAH >> Diglycolamine \approx Monoethanolamine >> Triethanolamine

During lapping, AHC compounds are believed to strongly chemisorb onto the AlTiC GMR Head specimen surfaces. As the ceramic material becomes abraded, the adsorbed AHC compounds impart a significant anionic charge to the swarf particles. Significant interparticle electrostatic repulsion facilitates rapid removal of the swarf from the specimen during lapping.

In order to minimize corrosion of the GMR Head metallic layers during lapping, it is desirable that the lapping fluid have an alkaline pH. Under alkaline conditions the surface of AlTiC acquires a net negative charge which would normally inhibit the adsorption of these anionic carboxylate compounds. The presence of hydroxyl groups on the citrate anion, however, promotes adsorption even under alkaline conditions. Such adsorption may result from the formation of highly stable cyclic ligands bound to surface metal cations or via strong hydrogen bonding interactions between the hydroxyl groups and the ceramic surfaces.

Formulations F and K showed enhanced MRRs during AlTiC Head Lapping. These rate enhancements likely result, in part, from the combination of pendent carboxylate and amide functional groups in the respective polymeric materials. The amide groups may promote polymer adsorption on the AlTiC surfaces via hydrogen bonding interactions.

A synergistic MRR effect is observed when lapping AlTiC with a Formulations E, I, and K. These Formulations include both citrate and polyaspartate species. Higher MRRs are observed by using formulations comprising a combination of these components compared to fluids containing either individual component alone. Applicant has discovered that preferred weight ratios of the polyaspartic (or aspartamide copolymer) to citrate salt range from 0.0 to about 1. In certain embodiments, Applicants' lapping formulation comprises a weight ratios of the polyaspartic (or aspartamide copolymer) to citrate salt range from about 0.1 to about 0.5.

Tables II and III recites Formulations L and M, respectively, comprising polyaspartate or aspartamide copolymer, in combination with a citrate blend.

Table II

FORMULATION L

Polyaspartamide Copolymer / Citrate Lapping Fluid Composition

Component	Concentration (Wt. %)
Distilled Water	90.7
MEA Citrate Tribasic	7.3
Poly Sodium Aspartate –co- diglycol Aspartamide (33 mol. % polymer repeat units amidated)	2.0

5

Table III

FORMULATION M

Polyaspartate / Citrate Lapping Fluid Composition

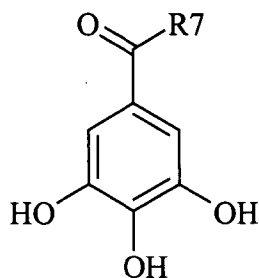
Component	Concentration (Wt. %)
Distilled Water	69.43
Triethanolamine Citrate Tribasic	25.9
Sodium Polyaspartate (Baypure DS 100)	4.67

10 In certain embodiments, Applicant's lapping composition comprises one or more species selected from the genus of compounds commonly known as flavonoids. Such flavonoids can be obtained from naturally-occurring materials, including without limitation green tea, brown tea, coffee, grapes, and the like. For example, grape extract is

also rich in polyphenolic flavonoids. As those skilled in the art will appreciate, such flavonoids comprise compounds having one or more benzene ring moieties with two or more hydroxyl groups attached to those aromatic ring structures.

In certain embodiments, Applicant's lapping composition includes vanillin and/or
5 vanillin derivatives made via reaction of vanillin's aldehyde moiety. Vanillin has been found to inhibit corrosion. In addition, the aldehyde group comprises functionality that allows vanillin to be grafted onto a polymer backbone via reaction with amines, i.e. formation of a Schiff Base, or reaction with a urea, or a reaction with other phenolic group moieties. As those skilled in the art will appreciate, Vanillin is nontoxic, has a
10 pleasant fragrance, and is available from both natural and synthetic sources. As those skilled in the art will further appreciate, Vanillin is structurally similar to catechol having two adjacent oxygen-containing groups attached on an aromatic ring, where one of those groups comprises a methyl ester.

In certain embodiments, Applicants' lapping composition further includes
15 compounds comprising hydroxyl substituted benzoic acids. In certain embodiments, Applicants' lapping composition is formed using Gallic Acid, Compound II where R7 is OH. In certain embodiments, Applicants' lapping composition includes Compound II wherein R7 is selected from the group consisting of OH, O-R16, N(R17)(R18), and the like, wherein R16 is selected from alkyl, alkali metal cation, alkaline earth cation, cation
20 IV, cation V, and the like, and wherein R17 and R18 are selected from alkyl, aryl, phenyl, and the like.



II

In certain of these embodiments, gallic acid is dispersed in distilled water at concentrations similar to those listed in Table I. Due to the highly acidic nature of Gallic acid, Applicant neutralizes the aqueous gallic acid solution using a variety of amines, alkali hydroxides, choline derivatives, guanidine derivatives, or TMAH prior to its use in lapping formulations. Suitable bases include but are not limited to alkanolamines, alkylamines, choline, guanidine derivatives, and the like. Alkaline lapping solutions are desirable to minimize corrosion which may occur during lapping of metallic surfaces. The reaction stoichiometry required to neutralize Applicant's gallic acid embodiments is a function of the pKa of the particular base used.

As a general matter, at least one equivalent of base is used for each equivalent of Gallic acid. As a further general matter, Applicant has found it desirable to have lapping fluid solutions comprising a pH greater than 8. As those skilled in the art will appreciate, such a resultant pH requires excess equivalents of base with respect to the gallic acid used.

For example, a 0.216 M aqueous solution of gallic acid was prepared having a pH of 3.05. Addition of 0.0429 mol. of ethanolamine to 200 ml of this solution corresponding to a 1:1 molar acid / base raised the pH to 6.89. Further ethanolamine was

added to give a 1.18 amine / acid molar ratio. This formulation had a clear green color with a pH of 8.72. Table IV lists the composition of this Formulation N.

Table IV

FORMULATION N

5

Gallic Acid Salt Lapping Fluid Composition

Component	Concentration (Wt.%)
Distilled Water	94.0
Ethanolamine	2.1
Gallic Acid	3.9

AlTiC bars were lapped using the Formulations recited in Table V under the same conditions as detailed in Table I above.

Table V

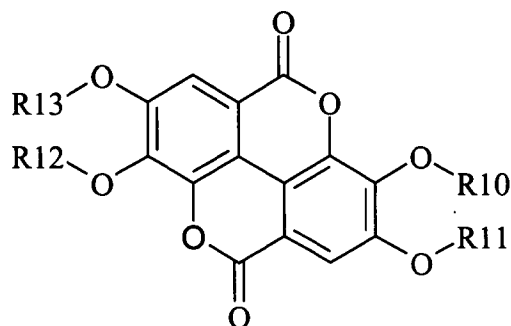
MRR Lapping Performance of Various Gallate-Derived Salts

Formulation	Component	Solution pH	Avg. MRR (g / min.)	MRR Relative to CONTROL
O	MEA Gallate	8.72	0.00093	233%
P	Potassium Gallate (1:1 mole ratio KOH/Gallic Acid)	8.98	0.00110	275%
Q	Potassium Gallate (3:1 mole ratio KOH/Gallic Acid)	10.1	0.00154	385%
R	Potassium Gallate (3:1 mole ratio KOH/Gallic Acid) AGED 48 HOURS	9.1	0.00181	453%
S	Potassium Gallate (Freshly Prepared)	9.1	0.00144	360%

Formulation R is formed using the same starting materials as Formulation Q.

Formulation R is aged, however, for 24 hours before use. During that 24 hour period, the
5 color of the solution changes from a clear, light green color to a very dark green color. In
addition, the pH of Formulation R is less than the pH of Formulation Q.

It is known in the chemical arts that Gallic acid dimerizes under aqueous alkaline
conditions to form Ellagic acid derivatives, Compound III, where R10, R11, R12, and
R13, are selected from the group consisting of ammonium salts, alkali metal ions,
10 alkaline earth ions, guanidinium ions, choline cations, and the like.



III

Gallic acid derived Formulations O through S are useful because the aromatic hydroxyls are deprotonated at alkaline pH and strongly adsorb upon the alumina surface. Applicant has found that citric acid derived formulations appear to be less strongly adsorbed at alkaline pH values compared to Gallic acid embodiments O through S.

In certain embodiments, Applicant's composition comprises polyphenolic compounds other gallic acid / gallates / gallamides. Such polyphenolic compounds include tannins; soluble black tea extracts; soluble green tea extract, such as for example Sunphenon 100S from Taiyo Kagaku Co. , Ltd.); quebracho bark extract used for leather tanning; tannic acid; humic acid; catechol; pyrogallol, d, l - Dopamine; d, l - DOPA derivatives; N-Acetyl Dopamine; and the like.

In certain embodiments, Applicant's composition comprises Catechin derivatives such as those found in tea extract, including but not limited to catechin, epicatechin, galocatechin, epigallocatechin, epicatechin gallate, galocatechin gallate, epigallocatechin gallate,

In certain embodiments, Applicant's composition includes Caffeoyl Derivatives, such as for example including esters, amides, and salts of caffeic acid. In certain

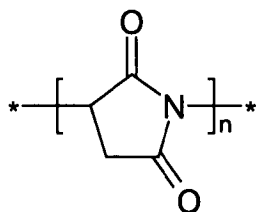
embodiments, Applicant's composition includes for example Procyanidin derivatives, Theaflavins, Thearubins, Phenolic Flavanol derivatives, Galloyl Glucose derivatives, Rugosin D, Enterochelin, Chlorogenic acid and its derivatives, Tyrosine derivatives, Alkyl Catechol derivatives including but not limited to tert-Butyl Catechol, and

5 combinations thereof.

In certain embodiments, Applicant's composition includes Phenol Ether derivatives including but not limited to guaiacol, veratrole, 2-methoxy phenol, mandelic acid, and the like, and combinations thereof. In certain embodiments, Applicant's composition includes one or more Orthoquinone derivatives.

10 Gallic acid, Ellagic acid, and related polyphenolic derivatives, exhibit low toxicity, and can be readily obtained via the hydrolysis of tannic acid, a natural product derived from wood by products. As those skilled in the art will appreciate, Tannic acid is essentially composed of a mixture of polysaccharides having a high concentration of gallic acid esterified along the saccharide polymer chains.

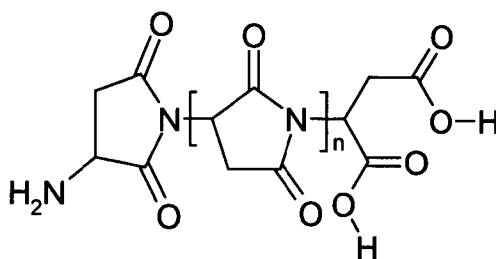
15 In certain embodiments, Applicants' lapping composition comprises polymeric materials. In certain of these polymeric materials embodiments, Applicant's polymeric materials include a plurality of pendent gallic acid moieties and/or gallic acid derivatives, such as without limitation gallamide moieties. In certain embodiments, these polymeric materials are formed by reacting a polymer which includes a plurality of polysuccinimide
20 repeat units VI with an amino-terminated gallamide.



VI

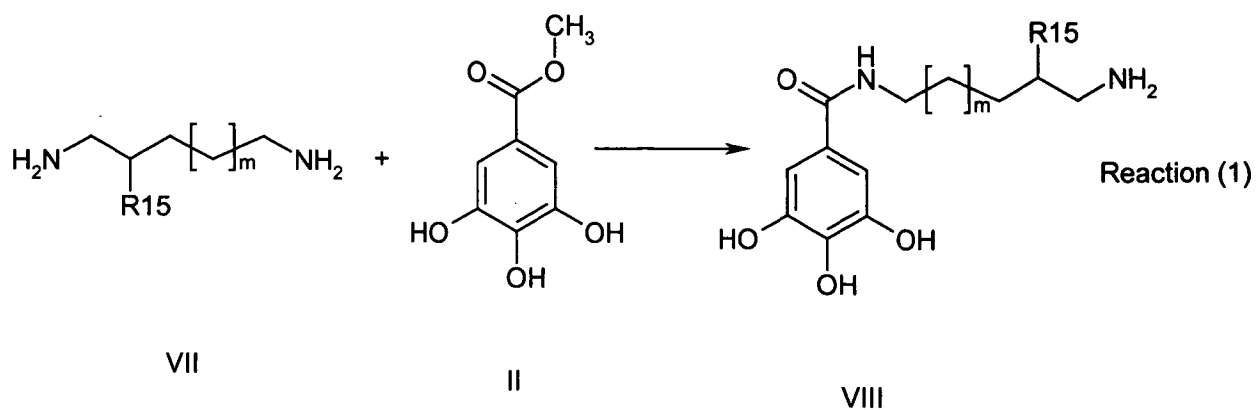
In certain embodiments, Applicant's polymeric material which includes a plurality of gallamide moieties is formed from polysuccinimide XI. In certain embodiments,

- 5 polysuccinimide XI has a number average molecular weight of about 3,000 Daltons.

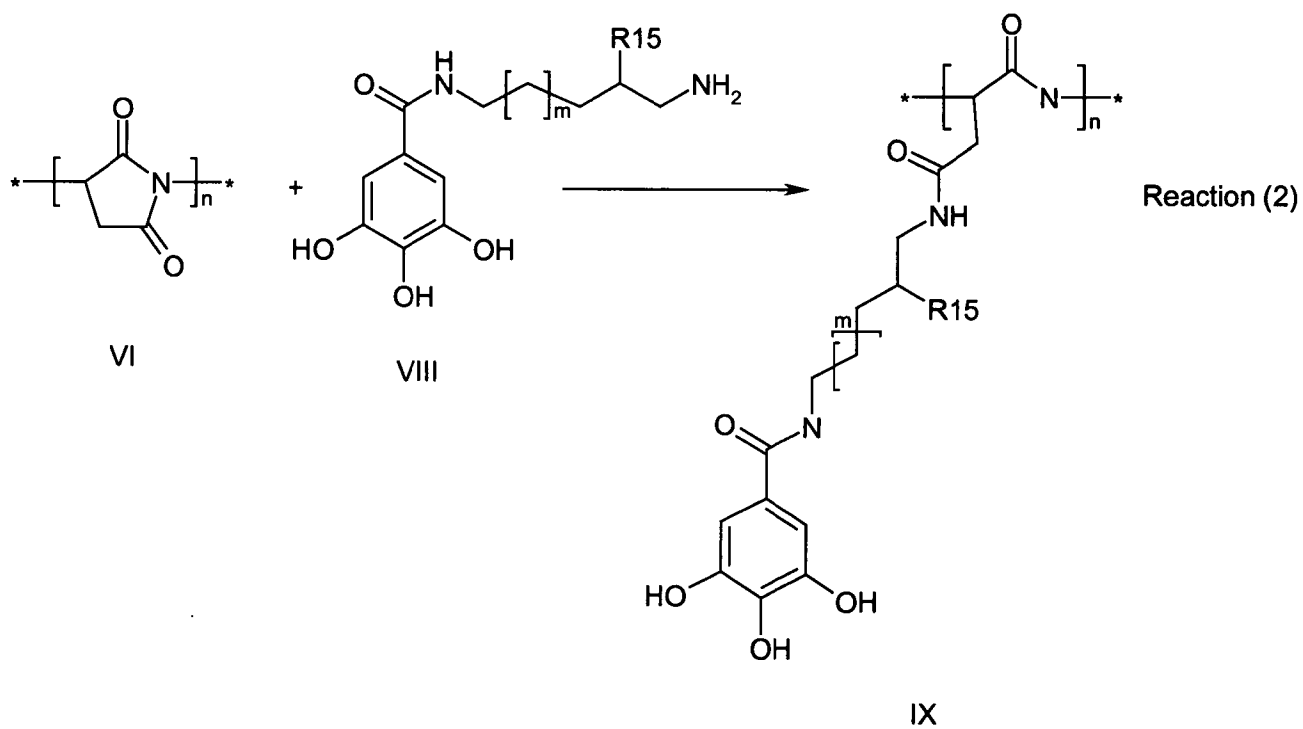


XI

In one embodiment, for example, methyl gallate, Compound II wherein R7 is O-Me, is reacted with diamine VII to give amino-terminated gallamide VIII wherein R15 is
 10 selected from the group consisting of hydrogen, alkyl, oxyalkyl, aryl, phenyl, and oxyphenyl, and wherein (m) is greater than or equal to 1 and less than or equal to about 10.



Amino-terminated gallamide VIII is then reacted with polysuccinimide VI to form derivatized polyaspartamide IX



EXAMPLE 1

Synthesis of Compound VIII

About 6.60 g (0.036 mole) of Methyl-3,4,5-trihydroxybenzoate was dissolved in about 46.4 g of Dimethyl Sulfoxide (DMSO) by gently heating the mixture at 45 °C for about 20 minutes with stirring. As those skilled in the art will appreciate, DMSO is a polar, aprotic solvent having low toxicity and good biodegradability. Other polar, aprotic solvents may be used in place of, or in combination with, DMSO. Such polar, aprotic solvents include, for example, N- Methyl Pyrrolidone, Sulfolane, Tetrahydrofuran, or Dimethyl Sulphone.

The resulting DMSO solution was then added to a 250 ml round bottom flask which had been previously flushed with inert nitrogen gas. About 10.368 g (0.08922 mole) of 2-methyl-1,5-pentanediamine, Compound VII, wherein R15 is methyl and (m) is 1, is sold in commerce by duPont under the tradename Dytex A, were then added to that DMSO solution, and the resulting reaction mixture was heated under vacuum at about 160 °C for about 8 hours. Diamine 2-methyl-1,5-pentanediamine was selected on the basis of its nucleophilicity combined with its high pK_{a1} value of 11.2.

During the reaction, methanol was removed from the reaction mixture as it was formed as the product from the aminolysis reaction occurring between the gallate ester and Diamine 2-methyl-1,5-pentanediamine. A clear, slightly reddish colored solution comprising about 26.6 weight percent compound VIII was obtained.

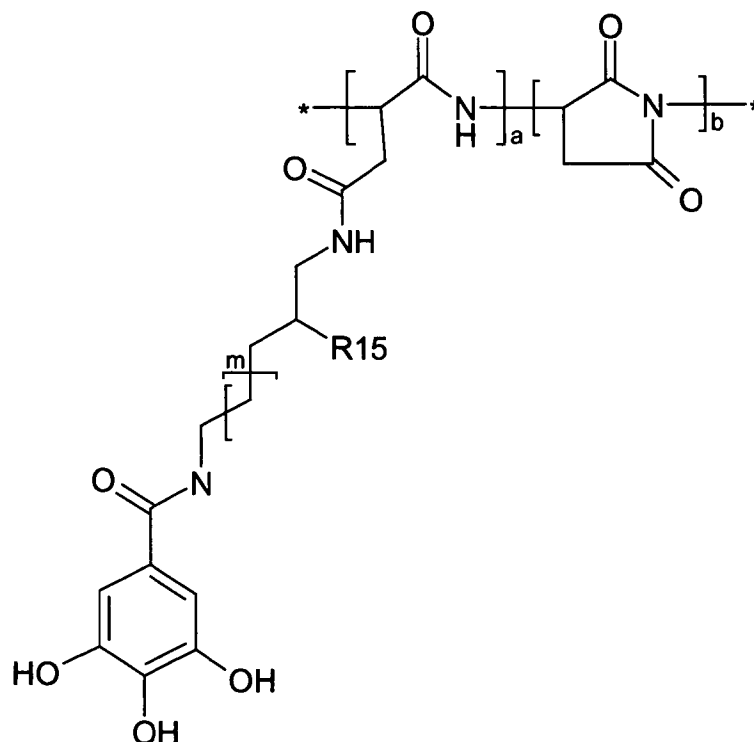
EXAMPLE 2

Polyaspartamide IX / X Preparation

About 7.82 g of 3,000 MW Polysuccinimide, polymer VI, sold in commerce by Bayer Chemicals Corp. Pittsburgh , PA under the tradename Baypure, was dissolved in
5 about 33.0 g DMSO at 55 °C. This yielded a solution comprising about 19.1 weight percent polymer VI

About 19.72 g of a DMSO solution of compound VIII, from Example 1, was diluted with about 8.61 g additional DMSO. This DMSO solution was rapidly mixed with the previously prepared DMSO / polymer VI solution.

10 In certain embodiments, a stoichiometric excess of Compound VII was added in an amount such that all the succinimide moieties were ring-opened. In other embodiments, less than a stoichiometric amount of VII was used, thereby giving co-polymer X having both succinimide linkages and substituted aspartamide linkages.



X

For example, in one embodiment about one-fourth of the succinimide repeat units in PSI were ring-opened using Reaction 2 to give co-polymer X wherein a is about 0.25(b). An exotherm was noticed upon mixing accompanied by a darkening of the solution such that it became sanguine in appearance. The solution was allowed to stand at room temperature for 30 minutes resulting in a clear, homogeneous, sanguine colored solution.

Applicant has discovered that grafting a plurality of gallamide groups onto a polyaspartic acid / aspartamide backbone is that aqueous solutions of the resulting product do not phase separate or precipitate upon standing. In contrast, the corresponding aqueous solutions of potassium gallate having similar molar concentration of gallate moieties darkens and phase separates as particulates upon standing. As noted

above, Gallic acid is known to undergo oxidative coupling when present in alkaline aqueous solutions...

Significantly, Applicant has found that attaching pendant gallamide groups to a polymer backbone allows the product to stay in solutions longer. In addition, Applicant
5 has found that using a polymer comprising pendent gallate / gallamide groups rather than a plurality of lower molecular weight gallate anions has the added advantage of forming electrosteric stabilized dispersions. Individual gallate anions comprise small discrete charges and impart electrostatic charges upon the swarf as it is removed during the polishing operation. Gallamide functionalized polymers, on the other hand also
10 electrostatically charge the particles but by virtue of them being attached to a backbone enables the adsorbed polymer chain to form a brush like structure upon the swarf; further hindering particulate agglomeration.

EXAMPLE 3

Lapping Solution Formulation

15 About 196.5 g of a solution comprising about 1.08 weight percent NaOH in water was combined with a DMSO solution of co-polymer X wherein a is about 0.25(b). The reaction mixture was stirred for about 1 hour at room temperature to give a solution having a pH of about 10.82. Table VI summarizes the composition of the aqueous lapping fluid prepared.

20 **TABLE VI**

Component	Concentration (Wt. %)
Sodium Polyaspartic-co-aspartamide	5.91

copolymer (25% pendant gallamide)	
DMSO	18.5
Distilled Water	75.59

AlTiC bars were then lapped using the method described about. An average MRR of 0.00133 g/min. was obtained using this lapping composition of Table VI.

The measured MRR for the composition of Table VI is significantly greater than the MRR of 0.00095 g/min. MRR measured when lapping AlTiC using a 25 weight percent solution of Baypure DS 100.

In other embodiments of Applicants' composition, pendant amine functionalized Gallamide VIII is reacted with other polymers to produce polymeric lapping fluid additive having both electrostatic and steric stabilization to polished swarf particles.

Such other polymers include, for example, maleic anhydride copolymers, such as Scripset 1000 – Polystyrene-co-maleic anhydride polymer obtained from Sartomer Company Inc. Exton, PA, or Gantrez AN - Polymethylvinylether-co-maleic anhydride obtained from International Specialty Products Inc. Wayne, NJ. In these embodiments, the amine functionalized gallamide VIII is reacted with the maleic anhydride copolymer in a polar aprotic solvent, followed by dissolution in aqueous alkaline solution to produce a polyelectrolyte lapping fluid additive.

In yet other embodiments, the amine functionalized gallamide VIII is with isocyanate prepolymers, acrylate oligomers, and/or epoxy oligomers, to produce water dispersible lapping fluid additives.

In other embodiments, polyethyleneimine (PEI) is reacted with methyl gallate to give a polymeric material comprising pendent gallamide moieties. As those skilled in the art will appreciate, PEI has a brush like structure, and is known in the art for its ability to serve as a dispersant in a wide variety of ceramic slurry formulations.

5 Applicant's gallamide functionalized PEI brush polymer is synthesized by adding methyl gallate to PEI either neat, or in a solvent, and allowing that reaction mixture to stand for several days at room temperature. During this reaction period, an aminolysis reaction occurs between the primary amine groups present on PEI and methyl gallate.

10 In certain embodiments, a stoichiometric excess of methyl gallate is used to react with essentially all of the amino moieties in the PEI polymer. Heating the reaction mixture in a nitrogen atmosphere at temperatures greater about 67 °C, i.e. the boiling point of methanol, drives this aminolysis reaction to completion. Aqueous solutions of these functionalized PEI polymers typically have pH values greater than about 9, and are suitable lapping fluids.

15 As those skilled in the art will appreciate, AlTiC comprises a two phase blend of Titanium Carbide, TiC, and Aluminum Oxide powders sintered together. TiC has an acidic isoelectric point (IEP) near pH 3.3. On the other hand, Aluminum Oxide is basic having an isoelectric point near pH 10. In water, AlTiC swarf comprises a mixture of the two particles. TiC particles acquire a negative electrostatic surface charge whereas
20 Aluminum Oxide particles acquire a positive surface charge. Due to mutual attraction between these two different kinds of charged particles, the swarf has a tendency to agglomerate.

To minimize, and hopefully prevent, swarf agglomeration, Applicant has found it desirable to impart identical surface charges to both the TiC particles and the Aluminum Oxide particles. Applicant has further found that citrate moieties and gallate moieties manifest an affinity, i.e. a selective adsorption, towards Aluminum Oxide particles and
5 TiC particles, respectively. Therefore, embodiments of Applicant's composition comprises blends of hydroxycarboxylate moieties, including without limitation citrate and/or polyaspartic acid - co- hydroxyethylaspartamide) anions, in combination with phenolate anions, including without limitation gallates, catecholates, pyrogallates, tannates, and the like. Applicant has found that such compositions comprising these
10 afore-described blends to be very useful aqueous lapping fluids for AlTiC, where those lapping fluids exhibit high Material Removal Rates.

EXAMPLE 4

A. Preparation Of Solution "A"

About 5.58 grams of anhydrous Citric acid were dissolved in about 96 grams of
15 distilled water. Thereafter, about 5.52 grams of monoethanolamine were added to that solution. This amount of MEA was sufficient to prepare a monoethanolammonium citrate tribasic species in solution.

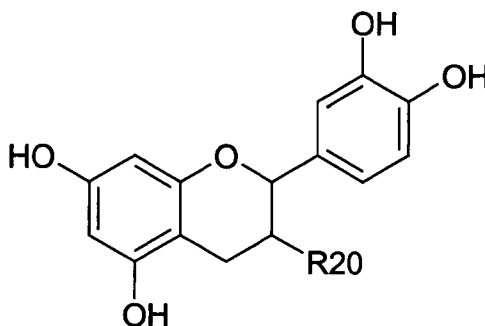
B. Preparation Of Solution "B"

About 5.48 grams of Gallic acid were added to about 96 grams of distilled water
20 followed by rapid addition of about 4.89 grams of KOH pellets with stirring.

C. Lapping Composition

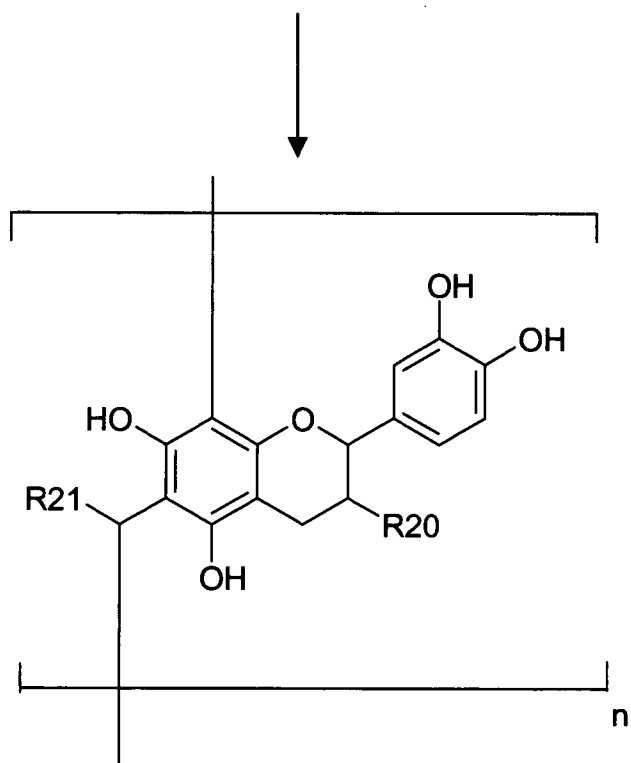
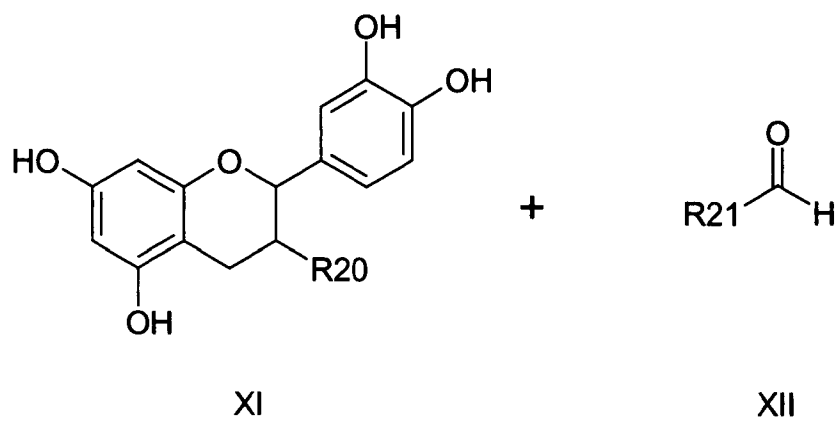
Solutions "A" & "B" were then combined to give a lapping composition comprising about a 50 / 50 mole mixture of citrate and gallate anions. AlTiC bars were then lapped using this solution and were found to have average Material Removal Rates of 0.002 g / min.

In certain embodiments, Applicant's lapping composition includes one or more catechin-aldehyde polymeric materials. Referring to Compound XI below, where R20 is OH, Compound XI comprises catechin or epi-catechin depending on the orientation of the R20 hydroxyl group.



XI

Reacting Compound XI with more than a two fold stoichiometric excess of aldehyde XII in acidic condition gives a polymeric material which includes the repeat unit XIII, wherein R21 is selected from the group comprising alkyl, phenyl, aryl, -COOH, -C(O)-Alkyl, hydroxyphenyl, and combinations thereof.



5

In certain embodiments, R₂₀ comprises OH. In other embodiments, R₂₀ comprises O-Galloyl, i.e. structure XIV.



10 (1997), which is hereby incorporated herein in its entirety.

10 (1997), which is hereby incorporated herein in its entirety.



In certain embodiments, Applicant's lapping composition includes Compound XV, wherein R20 and R21 are as described above. In certain embodiments, a plurality of moieties comprising Compound XV are reacted with Polymer XI to give a polymeric material comprising a plurality of pendent groups comprising Compound XV.